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IS 2267 (1995): Polystyrene Moulding and Extrusion Materials [PCD 12: Plastics]

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पोलियस्ट्रीन संचकन और बहिर्वेधन सामग्रियां – विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

POLYSTYRENE MOULDING AND EXTRUSION MATERIALS — SPECIFICATION

(Second Revision)

UDC 678·746·22·027·3·033·3

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 5

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Many outstanding features are responsible for making polystyrene (PS) one of the most popular Plastics. Since the basic material is colourless the polymer can be made crystal-clear or in an unlimited range of colours — from transparent to opaque. Excellent dimensional stability combined with negligible moisture absorption and freedom from warping or distortion within the thermal operating range make polystyrene useful where moulding to close tolerance is required. Its resistance to corrosive chemicals, such as acids (except oxidizing acids), alkalis, salts and lower alcohols is outstanding. Electrical properties are exceptionally good over the whole range of frequencies in practical use. The high-impact polystyrene or rubber modified polystyrene is characterized by improved shock resistance and increased elongation while still retaining reasonably good electrical and mechanical properties.

In the first revision of the standard a third type having medium impact strength was included. Further requirements for melt flow index, residual styrene, vicat softening point and colour fastness to artificial light were included while requirements for viscosity, volatile constituents, heat distortion temperature and water absorption were deleted.

In this second revision polystyrene materials are classified into groups according to classes and grades based on the basic property requirements (Table 1). It is not intended to imply that materials having the same designation give necessarily the same performance. Although the values listed are necessary to include the range of properties available in existing materials, users should not infer that every possible combination of properties exist or can be obtained.

This specification is, however, intended to be a means of calling out plastics materials used in the fabrication of end items or parts. Material selection should be made by those having expertise in the plastics field after careful consideration of the design and the performance required of the part, the environment to which it will be exposed, the fabrication process to be employed, the inherent properties of the material other than those covered by this specification, and the economics.

The properties included in the specification are those required to identify the compositions covered. There may be other requirements necessary to identify particular characteristics important to specialized applications. The purchaser should establish his own correlation between the properties of the processed articles and the material properties required for their achievement and these should be agreed between the purchaser and the supplier.

Properties of pigmented or coloured polystyrene materials can differ from the properties of natural or unpigmented polystyrene materials, depending on the choice of colourants and the concentration. If specific properties of pigmented polystyrene materials are necessary, prior testing between the materials supplier and end-user should be initiated.

Considerable assistance have been derived from ASTM D 4549-85 'Standard specification for polystyrene moulding and extrusion materials (PS)' issued by the American Society for Testing and Materials (USA) and ISO 1622/1-1985 'Plastics — Polystyrene (PS) moulding and extrusion materials — Part 1 : Designation' issued by the International Organization for Standardization (ISO), while preparing this standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

POLYSTYRENE MOULDING AND EXTRUSION MATERIALS — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements, methods of sampling and tests for polystyrene materials, both crystal and rubber modified, suitable for moulding and extrusion.

1.2 This standard establishes a classification for polystyrene (PS) materials which may be used as the basis for specification.

1.3 It does not apply to expanded polystyrene, styrene copolymers, homopolymers of substituted styrene. It applies to materials ready for normal use, unmodified and modified by colourants, additives, etc.

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provision of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standards are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
768 : 1982	Methods for evaluating change in colour (*first revision*)
2454 : 1985	Methods for determination of colour fastness of textile materials to artificial light (Xenon lamp) (*first revision*)
2530 : 1963	Methods of test for polyethylene moulding materials and polyethylene compounds
2828 : 1964	Glossary of terms used in the plastics industry
4486 : 1975	Recommended methods for the determination of permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including meter wavelengths
4905 : 1968	Methods for random sampling
8543 (Part 1/ Sec 2) : 1979	Methods of testing plastics : Part 1 Characterization of polymer structure and size, Section 2 Determination of density of solid plastics
8543 (Part 4/ Sec 1) : 1984	Methods of testing plastics : Part 4 Short term mechanical properties, Section 1 Determination of tensile properties
9833 : 1981	List of pigments and colourants for use in plastics in contact with foodstuffs, pharmaceuticals and drinking water
10142 : 1982	Styrene polymers for its safe use in contact with foodstuffs, pharmaceuticals and drinking water
13193 : 1992	Polyalkylene Terephthalates (PET and PBT) for moulding and extrusion
13360 (Part 1) : 1992	Plastics — Methods of testing: Part 1 Introduction
13360 (Part 2/ Sec 1) : 1992	Plastics — Methods of testing: Part 2 Sampling and preparation of test specimens, Section 1 Compression moulding test specimens of thermoplastics materials
13360 (Part 5/ Sec 13) : 1992	Plastics — Methods of testing : Part 5 Mechanical properties, Section 13 Rockwell hardness
13360 (Part 6/ Sec 1) : 1992	Plastics—Methods of testing : Part 6 Thermal properties, Section 1 Vicat softening temperature of plastics
13411 : 1992	Glass reinforced polyester dough moulding compounds

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 2828 : 1964 shall apply.

4 CLASSIFICATION

4.1 Polystyrene materials are classified into groups according to classes and grades as shown in the basic property table (Table 1).

NOTE — An example of this classification system is as follows:

The designation PS 111 would indicate, PS = Polystyrene;

1 (Group) = Crystal polystyrene;

1 (Class) = General purpose; and

1 (Grade) = Minimum-grade requirements as found in Table 1.

4.1.1 To facilitate the incorporation of future or special materials the other/unspecified category (0) for group, class and grade is shown in Table 1. The basic properties can be obtained from Table 2 or Table 3 as they apply (*see* **4.3**).

4.2 Reinforced and filled polystyrene materials are classified in accordance with Table 1 and Table 2. Table 1 specifies unreinforced material and Table 2, the properties after addition of reinforcement or lubricants at the normal levels indicated (*see* **4.2.1**).

4.2.1 *Reinforcements and Additive Materials*

A single letter will be used for the major reinforcement or combination or both, along with two digits that indicate the percentage of additives by mass with the tolerances tabulated as follows:

Symbol	*Material*	*Tolerance (Based on Total Mass)*
C	Carbon and graphite fibre reinforced	± 2 percent
G	Glass	± 2 percent
M	Mineral-reinforced	± 2 percent
L	Lubricants	By an agreement between the supplier and the user
R	Combination of reinforcement or fillers or both	do

NOTE — This part of the system uses the type and percentages of additive to designate the modification of the basic material. Percentage of additives can be shown on the supplier's Technical Data Sheet unless it is proprietary. If necessary, additional requirements shall be indicated.

4.2.2 *Detail Requirements*

An identifying number is made up of the letter A and five digits comprising the cell numbers for the new requirements in the designated order as they appear in Table 2.

4.2.2.1 Although the values listed are necessary to include the range of properties available in existing materials, users should not infer that every possible combination of the properties exists or can be obtained.

Example : The designation PS 110 G 15 A 12332 would indicate the following material requirements from Table 2 :

PS 110	Polystyrene crystal from Table 1;
G 15	Glass-reinforced at 15 percent nominal;
A	Table 2 for property requirements
1	Tensile strength, 510 kg/cm^2, *Min*;
2	Percent elongation, 10, *Min*;
3	Izod impact, 4 kg. cm/cm notch, *Min*;
3	Deflection temperature, 102°C, *Min*; and
2	Specific gravity, 1·2, *Min* .

4.3 Table 3 has been incorporated into this specification to facilitate the classification of special materials where Table 1 or Table 2 does not reflect the required properties. Table 3 shall be used in the same manner as Table 2.

Example : The designation PS 110 B 76013 would indicate the following, with the material requirements from Table 3 :

PS 110 = Polystyrene, crystal, from Table 1;

B = Cell Table 3 for property requirements;

7 = Tensile strength, 450 kg/cm^2, *Min*;

6 = Percent elongation, 30, *Min*;

0 = Unspecified Izod impact;

1 = Vicat softening temperature, 85°C, *Min*; and

3 = Melt flow rate, 3·0, *Min*.

5 REQUIREMENTS

5.1 General

The material composition shall be uniform and shall conform to the requirements specified herein. The colour and form of the materials shall be agreed upon by the supplier and the user. Specification changes due to the effects of colourants shall be noted by both parties.

NOTE — Properties of pigmented or coloured PS materials can differ from the properties of the natural or unpigmented PS material, depending on the choice of colourants and the concentration. If specific properties of pigmented PS materials are necessary, prior testing between the material supplier and end user should be initiated.

5.2 Basic Requirements

Basic requirements from Table 1, as they apply, are always in effect unless these requirements are superseded by specific suffix requirements (Table 2 or Table 3), which always take precedence.

5.3 Detail Requirements

Test specimens for the various materials shall conform to the requirements prescribed in Tables 1, 2, and 3 as they apply.

Observed or calculated values obtained from analysis, measurements or test shall be compared, after rounding off in accordance with

Table 1 Polystyrene Materials, Detail Requirements, Natural Colour Only

(*Clause* 4.1)

Group	Description	Class	Description	Grade	Description1)	Melt Flow Rate (200°/5 kg), g/10 min, *Min*	Izod Impact2) (12·7 × 6·35 mm) kg. cm/cm Notch, *Min*	Tensile Strength3),4),5) at Yield, kg/cm^2, *Min*	Percent, Elongation *Min* 4),5)	Vicat Softening Point (Method B) °C, *Min*
1	Crystal	1	General purpose	1		3·0	2·0	450	—	100
				2		8·0	1·5	400	—	95
				3		15·0	1·5	370	—	90
		0	Other	0	Other	Unspecified	Unspecified	Unspecified	Unspecified	Unspecified
2	Rubber modified	1	Medium impact	1		5·0	5·0	300	25	95
				2		6·0	4·0	320	20	90
				3		7·0	3·0	350	10	90
				0	Other	Unspecified	Unspecified	Unspecified	Unspecified	Unspecified
		2	High impact	1		4·0	8·0	220	40	90
				2		5.0	7·0	250	30	90
				3		6·0	6·0	270	25	90
				0	Other	Unspecified	Unspecified	Unspecified	Unspecified	Unspecified
		3	Super-high impact	1		3·0	10·0	220	45	95
				2		4·0	9·0	250	40	90
		0	Other	0	Other	Unspecified	Unspecified	Unspecified	Unspecified	Unspecified
0	Other	0		0	—	—	—	—	—	—

1)No descriptions are listed unless needed to describe a special grade under the class. All other grades are listed by requirements.

2)kg. cm/cm Notch = ft./lbf/in. × 5·44, ft. lbf/in Notch = 53·39 J/m.

3)kgf/cm^2 = psi × 0·070 4 : 6.986 5 × 10^{-3} psi = MPa.

4)Values in this column for crystal PS are not yield, but break strengths; crystals PS do not exhibit a yield point.

5)Tensile properties determined on 3·2 mm thick injection moulded type 1 specimen tested at 5 mm/min.

Table 2 Reinforced Polystyrene Material, Detail Requirements

(*Clauses* 4.1.1 *and* 4.2)

Designation Order Number	Property	0	1	2	3	4	5	6	7	8	9
1	Tensile strength, kg/cm^2 *Min*1)	Unspecified	510	610	715	815	920	1 020	1 120	1 225	To specify
2	Percent elongation, *Min*	do	5	10	15	20	25	30	35	40	do
3	Izod impact (12·7 × 6·35 mm), kg.cm/cm Notch, *Min*2)	do	1	2	4	4	6	8	10	12	do
4	Deflection temperature under flexural load (DTUL) (Annealed3)) 1·82 MPa, °C, *Min*	do	94	98	102	106	110	114	118	122	do
5	Specific gravity, *Min*	do	1·1	1.2	1·4	1·6	1·8	2·0	2·2	2·4	do

1)kg/cm^2 × 14·20 = psi; MPa × 145 = psi.

2)kg.cm/cm × 0·183 8 = ft. lbf/in.; J/m × 18·73 × 10^{-3} = ft. lbf/in.

3)Deflection temperature under flexural load (12·7 × 3·2 mm) sample.

Table 3 Unreinforced Polystyrene Material, Detail Requirements

(*Clauses* 4.1.1 *and* 4.3)

Designation Order Number	Property	0	1	2	3	4	5	6	7	8	9
1	Tensile strength, kg/cm^2, *Min*1)	Unspecified	150	200	250	300	350	400	450	550	To specify
2	Percent elongation, *Min*	do	5	10	15	20	25	30	35	40	do
3	Izod impact (12·7 × 6·35 mm), kg.cm/cm Notch. *Min*2)	do	1	2	4	6	8	10	15	20	do
4	Vicat softening point (Method B), °C, *Min*	do	85	90	95	100	105	110	115	120	do
5	Melt flow rate (200°C/ 5 kg), g/10 min, *Min*	do	0·5	1·0	3·0	5·0	7·0	9·0	12·0	15·0	do

1)kg/cm^2 × 14·20 = psi; MPa × 145 = psi.

2)kg.cm/cm × 0·183 8 = ft. lbf/in.; J/m × 18·73 × 10^{-3} = ft. lbf/in.

IS 2 : 1960, directly with the specified limiting value. Conformance or non-conformance with the specification is based on this comparison.

5.4 Additional Requirements

Other properties to be selected shall be determined by the characteristics required for processing and the characteristics required of the processed article.

NOTE — The purchaser should establish his own correlation between the properties of the processed article and the material properties required for their achievement.

5.4.1 Typical additional properties to be measured are as follows:

a) Colour fastness to artificial light;
b) Particle size;
c) Flammability, cm/min;
d) Rockwell hardness;
e) Power factor at 1 MHz;
f) Permittivity at 1 MHz; and
g) Residual styrene, percent by mass.

5.4.2 *Contact with Foodstuffs, Pharmaceuticals and Drinking Water*

Where the products are used in contact with foodstuffs, pharmaceuticals and drinking water, its requirements with respect to the material shall also be met as per IS 10142 : 1982.

In case coloured materials are used for food contact applications, it shall also comply with the lists and limits of the pigments and colourants prescribed in IS 9833 : 1981.

6 PREPARATION OF TEST SPECIMENS

The test specimens shall be moulded by injection or compression moulding process as specified in IS 13360 (Part 2/Sec 1) : 1992, for the specific products, or they shall be moulded as specified by the resin supplier.

7 CONDITIONING

Test specimens shall be conditioned in standard atmospheric condition of 27 ± 2°C and 65 ± 5 percent relative humidity in accordance with IS 13360 (Part 1) : 1992 before performing the required tests.

Tests shall be conducted in the standard atmospheric condition of 27 ± 2°C and 65 ± 5 percent relative humidity.

8 TEST METHODS

Unless otherwise stated or unless other test methods are agreed to by the user and the supplier the various properties shall be determined in accordance with the test methods given as follows:

Sl No.	*Characteristics*	*Test Methods, Ref to Clauses of IS*	*Annex of this Standard*
i)	Melt flow rate (MFR) g/10, min at 200°C/5 kg	7 of IS 2530 : 1963	—
ii)	Izod impact (Notched), J/m	Annex E of IS 13411 : 1992	—
iii)	Tensile strength, kgf/cm^2	IS 8543 (Part 4/Sec 1) : 1984	—
iv)	Elongation, percent	do	—
v)	Deflection temperature under load, °C	Annex H of IS 13411 : 1992	—
vi)	Specific gravity	IS 8543 (Part 1/Sec 2) : 1979	—
vii)	Vicat softening point	IS 13360 (Part 6/Sec 1) : 1992 (Method B)	—
viii)	Colour fastness to artificial light	—	Annex A
ix)	Particle size	—	IS 13360 (Part 3/Sec 8) is under preparation, till such time ASTM D 1921-1989 shall be followed
x)	Flammability, cm/min	Annex C of IS 13193 : 1992	—
xi)	Rockwell hardness	IS 13360 (Part 5/Sec 13) : 1992	—
xii)	Power factor	IS 4486 : 1975	—
xiii)	Permittivity	do	—
xiv)	Residual styrene	—	Annex B

9 SAMPLING

Unless otherwise agreed upon between the user and the supplier, the materials shall be sampled in accordance with Annex C. Adequate statistical sampling shall be considered as an acceptable alternative. A lot of resin shall be considered as a unit of manufacture as prepared for shipment, and may consist of a blend of low or more production runs or batches of material.

10 PACKING AND MARKING

10.1 Packing

The material shall be packed in well-closed containers, as agreed to between the purchaser and the supplier.

10.2 Marking

Each container and package shall be clearly marked with the following:

a) Indication of the source of manufacture and trade-mark, if any;
b) Material designation:
c) Net mass;
d) Month and year of manufacture; and
e) Batch No. or Lot No.

10.3 BIS Certification Marking

The containers and packages may also be marked with the Standard Mark.

10.3.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

ANNEX A

(*Clause* 8)

TEST FOR COLOUR FASTNESS TO ARTIFICIAL LIGHT

A-1 GENERAL

A-1.1 Outline of the Method

A sheet moulded from the material and the standard pattern for light fastness rating are exposed to artificial light for stipulated period and colour compared.

A-2 APPARATUS

A-2.1 Artificial Light Source

Either a xenon arc with filter or an enclosed carbon arc, such that no part of the spectrum of the radiation shall have a wave length less than 2 900°A.

A-2.2 Geometric Grey Scale

For assessing the change in colour (*see* IS 768 : 1982).

A-2.3 Standard Patterns

A set of 8 standard patterns of blue wool cloth for light fastness rating, as prescribed in IS 2454 : 1985.

A-2.4 Thermometer

The black panel thermometer unit consisting of stainless steel panel, 70 mm × 150 mm × 0·9 mm, to which is mechanically fastened a stainless steel bimetallic dial-type thermometer. The thermometer shall have a stem 4 mm in diameter with 45-mm dial. The sensitive portion extending 38 mm from the end of the stem shall be located in the centre of the panel, 64 mm from top end 48 mm from bottom of the panel. The face of the panel with thermometer stem attached shall be finished with two coats of light and water-resistant black enamel.

A-3 TEST SPECIMENS

The specimens shall be 40 mm × 25 mm sheets moulded suitably from the material as prescribed in **6**.

A-4 PROCEDURE

A-4.1 Cut out sufficient number of test specimens. Cover half of the portion of each test specimen in a manner to protect the covered area from light. This may be done by covering the half portion with black paper. The whole set of test specimens shall be pinned to piece of cardboard.

A-4.2 The set of 8 standard patterns of size 30 mm × 5 mm shall be pinned to another piece of cardboard and half the portion of each standard covered with black paper.

A-4.3 Expose the test pieces and the standard to the artificial light source. The temperature of air surrounding the test specimens during test and assessment shall be not more than 60°C when measured by black panel thermometer. Observe the test pieces and standards at suitable intervals until standard pattern for light fastness rating No. 5 shows fading equivalent to Grade 4 on geometric grey scale. Compare the exposed and unexposed portions of test specimens with the standard pattern for light fastness rating No. 5.

ANNEX B

(*Clause* 8)

DETERMINATION OF RESIDUAL STYRENE MONOMER

B-1 PRINCIPLE

Dissolution of the polymer sample in chloroform or dichloromethane. Precipitation of the polymer by the addition of methanol and injection of a small volume of the remaining solution into a gas chromatograph to obtain separation of styrene and other volatile aromatic hydrocarbons. The solvent contains a known amount of *n*-butylbenzene as an internal standard for quantitative evaluation.

B-2 REAGENTS

B-2.1 Chloroform or Dichloromethane, analytical grade. A small amount of ethanol which may be contained in the chloroform for stabilization does not affect the determination.

B-2.2 Methanol, analytical grade.

B-2.3 *n*-Butylbenzene, of sufficient purity for analytical use.

B-2.4 Styrene and (if required) other aromatic hydrocarbons such as ethylbenzene, cumene or alpha-methylstyrene of sufficient purity for calibration. Styrene must be freshly distilled and stored at 0°C until required for use. When mixed with an equal volume of methanol, the styrene shall give a clear mixture.

B-2.5 Polyethylene Glycol, molecular mass 15 000 to 20 000 (Carbowax 15 to 20 M has been found satisfactory.)

B-2.6 Diatomaceous Earth, particle size 210 to 250 μm. (Celite, acid-washed grade has been found satisfactory.)

B-2.7 Helium, Hydrogen, and air as carrier and fuel gases for gas chromatography. For use with the flame ionization detector, nitrogen may be applied as the carrier gas instead of helium.

NOTE — Changes in carrier gas are permissible only if this has proved to give the same results.

B-3 APPARATUS

B-3.1 Gas Chromatograph, with injection port for liquid sample, flame ionization detector, and recorder. Some other type of ionization detector may be used, and, in exceptional cases, a thermal conductivity detector. The latter, however, entails lower sensitivity.

B-3.2 Micro-Syringe, 150 μl.

B-3.3 Analytical Balance

B-4 PREPARATION OF SAMPLES

The sample may be taken from material in the form of a powder, pellets or moulding. Large pieces shall be reduced to fragments sufficiently small for weighing a sample amount as close as possible to 1·5 g.

B-5 PROCEDURE

B-5.1 Preparation of Solvent for the Sample

Weigh 900 ± 1 mg of *n*-butylbenzene (**B-2.3**) into a 25 ml flask, transfer quantitatively into a 1 000 ml volumetric flask, and dilute to 1 000 ml with chloroform or dichloromethane (**B-2.1**). During the dilution process, maintain the liquid at a temperature of 20 ± 0·5°C.

B-5.2 Preparation of the Sample Solution

Weigh to the nearest 1 mg, 1·5 g of the polymer sample and transfer to a 100 ml conical flask, preferably with a serum cap closure. Add from a pipette exactly 10 ml of the solvent prepared according to **B-5.1** and kept at 20 ± 0·5°C. After closing the flask, allow the polymer to dissolve, with shaking if necessary. After complete dissolution, add exactly 5 ml of methanol (**B-2.2**) kept at 20 ± 0·5°C, either by syringe a or from a pipette. After vigorous shaking, allow the precipitation to settle. For injection into the gas chromatograph, extract required the amount with the syringe (**B-3.2**) from the supernatant solution.

B-5.3 Preparation of Calibration Solutions

Prepare solution for calibration by weighing 600 ± 0·5 mg of *n*-butylbenzene together with an appropriate amount of styrene or (if required) of another hydrocarbon to be determined, and diluting in a volumetric flask to 1 000 ml with a mixture of chloroform for (dichloromethane) and methanol in a volume ratio of 2 : 1. Appropriate amounts of hydrocarbon are recommended as follows:

- 100, 200, 500, 800 and/or 1 000 mg of styrene;
- 10, 20, 50, 80 and/or 100 mg of other hydrocarbons.

All hydrocarbons are to be weighed to the nearest 0·5 mg. The temperature of the liquids should be 20 ± 0·5°C. A separate calibration solution, or a series of calibration solutions, is to be prepared for each single hydrocarbon to be determined. The solutions are kept for injection into the gas chromatograph.

B-5.4 Gas Chromatographic Procedure

B-5.4.1 *Gas Chromatographic Operating Conditions*

Column : metal or glass tubing,4 ·5 m (15 ft) length, 4 mm internal diameter, is recommended. The column is packed with a support medium of diatomaceous earth (**B-2.6**) which is coated with 10 percent (m/m) polyethylene glycol (**B-2.5**).

NOTE — Methods of coating and packing are not specified, but may be chosen so as to obtain satisfactory separating efficiency of the column. The column shall be aged for 24 h at 150°C with gas flow. Changes in column materials or column dimensions are permissible only if this has proved to give the same results.

Column temperature : 80°C isothermal.

Temperature of injection port : 150°C.

Temperature of detector compartment : 150°C.

Carrier gas : Helium for nitrogen under the conditions indicated in **B-2.7** .

Flow rate of carrier gas : 100 cm^3/min.

Flame ionization detector, in which the hydrogen and air flow rates are adjusted to give:

a) high sensitivity of response;

b) linear response over the range of concentration being measured; and

c) only insignificant effect of small changes in flow rates on response or sensitivity.

Recorder speed : 0·5 cm/min (0·2 in/min) is recommended.

B-5.5 Gas Chromatographic Recording of Sample and Calibration Solutions

According to the sensitivity of the gas chromatograph used, inject a suitable volume of the sample solution (prepared according to **B-5.2**) or the calibration solution (prepared according to **B-5.3**). The inject volume is not critical for the calculation of results, but shall be identical for corresponding sample and calibrations solutions.

Develop the gas chromatogram until the *n*-butylbenzene is completely eluted, or, if hydrocarbons with higher retention times are to be determined, until complete elution of these.

B-5.6 Evaluation of Gas Chromatographic Peaks

The retention times of styrene, *n*-butylbenzene, and (if required) of other hydrocarbons to be determined must be known, at least relative to each other.

NOTE — In the following table approximate values of the retention time are given for some of the most frequently occurring components. The exact values vary according to the density of the column packing, the age of the column, and other gas chromatographic parameters:

Component Hydrocarbons	*Retention Time*, min	*Retention Time Relative to n-butylbenzene*
Ethylbenzene	9	0·44
Cumene	10·5	0·51
n-Propylbenzene	12·5	0·61
Styrene	16·5	0·80
n-Butylbenzene (standard)	20·5	1
α-Methylstyrene	25	1·22

Other components which might occur in smaller amounts are benzene toluene, *m*- and *p*-xylene, *o*-, *m*- and *p*-ethyltoluene and sec-butylbenzene.

The peak areas of the components to be determined and of *n*-butylbenzene are determined by:

a) electronic integration; or

b) area estimation on the basis of the formula:

area (A) = peak height × halfwidth; or

c) planimetry.

Use of Method 2 is recommended only for peaks with a horizontal base line and having a shape as close as possible to that of an isosceles triangle, in order to minimize the inaccuracy of measurement. In most cases, Method 2 is only applicable to the peaks of the major components, that is, styrene and *n*-butylbenzene. For routine determinations, peak height measurements of both sample and calibration chromatograms will be sufficient. The method of area evaluation chosen must be identical for corresponding peaks of sample solution and calibration solution.

B-6 EXPRESSION OF RESULTS

B-6.1 Calculation of Results from a Calibration Graph

If several calibration solutions with different concentrations of one single aromatic hydrocarbon are available, a calibration graph is drawn by plotting the ratios of peak areas, A'_a/A'_s, against the respective concentrations in milligrams per millilitre. With the corresponding ratios determined from the sample solution, A_a/A_s, the concentration of the substance to be determined in the sample solution, C_a, may be taken from the calibration graph. From C_a, the percentage (m/m) of aromatic hydrocarbon in the polystyrene sample, P_a, is calculated according to the relation:

$$P_a = \frac{1{\cdot}5\, C_a}{m_p}$$

Symbols used :

A'_a	is the peak area of styrene or other hydrocarbon to be determined in the calibration solution;
A'_s	is the peak area of the internal standard (*n*-butylbenzene) in the calibration solution;
A_a	is the peak area of styrene or other hydrocarbon to be determined in the sample solution;
A_s	is the peak area of the internal standard (*n*-butylbenzene) in the sample solution;
C_a	is the concentration of styrene or other hydrocarbon in the sample, expressed in milligrams per millilitre;
m_p	is the mass of the polymer sample, expressed in grams;
P_a	is the content of styrene or other volatile aromatic hydrocarbon compound in the sample, expressed in percent (m/m).

If m_p amounts to exactly 1·5 g, as specified in this method, the numerical value of C_a as taken from the calibration graph is identical to the numerical value of P_a.

B-6.2 Calculation of Results from a Single Point Calibration

Provided a linear relationship exists between peak areas and respective concentrations of styrene or other hydrocarbons determined, the following relation may be applied:

$$P_a = 1{\cdot}5\, \frac{m'_a\, A_a/A_s}{m_p\, A'_a/A'_s}$$

where

A'_a , A'_s , A_a , A_s , m_p and P_a are as in **B-6.1**; and

m'_a is the mass of styrene or other hydrocarbon weighed into 1 000 ml calibration solution (*see* **B-5.3**).

NOTE — If the internal standard is present as a component in the polymer, as may be seen from a blank run of the sample solution, that is, without internal standard added, then the monomer content may be determined by the internal addition method.

B-6.3 Precision of Measurement and Sensitivity

The dispersion of results on repeated measurement of one aromatic hydrocarbon, for example, styrene, in the sample may not exceed 0·01 units of P_a around the arithmetic mean.

A lower limit of detection of the order of 10 ppm may be expected from the method.

B-7 TEST REPORT

The test report shall include the following particulars:

a) Type and identification characteristics of the polymer tested;

b) Any deviation of the gas chromatographic equipment and procedure from the standard equipment with flame ionization detector and procedure described herein; and

c) Content of styrene monomer (and other volatile aromatic hydrocarbons, if determined) expressed as a percentage by mass of the polymer sample and rounded off to the nearest 0·01 percent.

ANNEX C

(*Clause* 9)

SAMPLING OF POLYSTYRENE MOULDING MATERIALS

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.1 Sample shall not be taken in an exposed place.

C-1.2 The sampling instrument, which shall be made of glass, stainless steel or any other material on which polystyrene moulding material has no action, shall be clean and dry.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers selected for sampling from adventitious contamination.

C-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-1.5 The samples shall be placed in suitable, clean, dry, air-tight sheet metal or glass containers on which the material has no action.

C-1.6 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, the date of sampling and the month and year of manufacture of the material.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

C-2.2 A number of containers, consisting 10 percent of the containers in a lot but not less than 3 containers in any case, shall be selected at random from a lot for the purpose of drawing samples for test (*see* IS 4905 : 1968).

C-3 TEST SAMPLES AND REFEREE SAMPLE

C-3.1 Preparation

To prepare a set of test samples, draw with an appropriate sampling instrument, from freshly opened containers which have been selected for sampling, an equal number of scoopfuls of material from any point at least 75 mm below the surface and 75 mm above the bottom of large containers, and from any point at least 25 mm below the surface and 25 mm above the bottom of small containers. The sample prepared by mixing the portions from each container shall be not less than eight times the quantity which is estimated to be required for carrying out all the tests. Divide this composite sample into the required number of reduced samples. Each set of these reduced samples shall constitute the test sample.

C-3.2 Three sets of test samples, each not less than twice the quantity required for the purpose of testing, representative of each selected container (*see* **C-3.1**) shall be transferred immediately to thoroughly dried containers, which shall be sealed airtight with an appropriate stopper. These containers shall be marked with all the particulars of sampling given under **C-1.6**. One set of the test samples shall be sent to the purchaser and one to the supplier.

C-3.3 Referee Sample

The third set of the test samples, bearing the seals of the purchaser and the supplier shall constitute the referee sample, to be used in case of dispute between the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier.

C-4 TEST FOR ACCEPTANCE

C-4.1 Examination and Tests

The purchaser may examine and test separately samples from each of the lots (*see* **C-2.1**) for compliance with the requirements of the standard, or he may prepare for the purpose of such examination and at any stage of the progress of the examination, a composite sample representing the whole of the consignment, by mixing the test samples.

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